UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

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VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**FIELD PORTABLE X-RAY FLUORESCENCE ANALYZER**
APPLICATION:	**MEASUREMENT OF METALS IN SOIL**
TECHNOLOGY NAME:	**X-MET 920-P AND X-MET 940**
COMPANY:	**METOREX, INC.**
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The U.S. Environmental Protection Agency (EPA) has created a program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the Environmental Technology Verification (ETV) Program is to further environmental protection by substantially accelerating the acceptance and use of improved and more cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of the Metorex X-MET 920-P and 940 analyzers.

PROGRAM OPERATION

The EPA, in partnership with recognized testing organizations, objectively and systematically evaluates the performance of innovative technologies. Together, with the full participation of the technology developer, they develop plans, conduct tests, collect and analyze data, and report findings. The evaluations are conducted according to a rigorous demonstration plan and established protocols for quality assurance. The EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, selected PRC Environmental Management, Inc., as the testing organization for the performance verification of field portable X-ray fluorescence (FPXRF) analyzers.

DEMONSTRATION DESCRIPTION

In April 1995, the performance of seven FPXRF analyzers was determined under field conditions. Each analyzer was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Standard reference materials (SRM) and performance evaluation (PE) samples also were used to independently assess the accuracy and comparability of each instrument.

The demonstration was designed to detect and measure a series of inorganic analytes in soil. The primary target analytes were arsenic, barium, chromium, copper, lead, and zinc; nickel, iron, cadmium, and antimony were secondary analytes. The demonstration sites were located in Iowa (the RV Hopkins site) and Washington (the ASARCO site). These sites were chosen because they exhibit a wide range of concentrations for most of the target metals and are located in different climatological regions of the United States; combined, they exhibit three distinct soil types: sand, clay, and loam. The conditions at these sites are representative of those environments under which the technology would be expected to operate. Details of the demonstration, including a data summary and

discussion of results, may be found in the report entitled "Environmental Technology Verification Report, Field Portable X-ray Fluorescence Analyzer, Metorex X-MET 920-P and 940." The EPA document number for this report is EPA/600/R-97/146.

The EPA Method 6200 was tested and validated using the data derived from this demonstration. This method may be used to support the general application of FPXRF for environmental analysis.

TECHNOLOGY DESCRIPTION

These analyzers operate on the principle of energy dispersive X-ray fluorescence spectroscopy where the characteristic energy components of the excited X-ray spectrum are analyzed directly by an energy proportional response in an X-ray detector. Energy dispersion affords a highly efficient, full-spectrum measurement which enables the use of low intensity excitation sources (such as radioisotopes) and compact battery-powered, field-portable electronics. The FPXRF instruments are designed to provide rapid analysis of metals in soil. This information allows investigation and remediation decisions to be made on-site and reduces the number of samples that need to be submitted for laboratory analysis. In the operation of these instruments, the user must be aware that FPXRF analyzers do not respond well to chromium and that detection limits may be 5 to 10 times greater than conventional laboratory methods. As with all field collection programs, a portion of the samples should be sent to a laboratory for confirmatory analyses.

Metorex considers the X-MET 920-P and 940 to have equivalent performance characteristics. Advances in electronics have led to the redesign of the 920-P into a smaller and lighter version, the X-MET 940. At the time of the demonstration, the 920-P was commercially available while the 940 was tested as a prototype.

These instruments are designed to produce quantitative data on the concentration of metals in soils, sludges, and other solids. Each instrument consists of a battery-operated electronics unit and a solid-state probe system (SSPS). The SSPS houses two excitation sources and a lithium-drifted (Si[Li]) detector (cooled by liquid nitrogen) for elemental excitation and detection. The SSPS allows for *in situ* analysis or the measurement of samples in cups. Either instrument can be operated and calibrated using site-specific calibration samples or through the use of fundamental parameters (FP) calibration software. During this demonstration, the FP software was used and fine-tuned with the use of one site-specific sample to improve data comparability. During this demonstration, the instruments were configured to report arsenic, barium, cadmium, copper, chromium, iron, lead, nickel, and zinc. At the time of the demonstration, each instrument cost about $55,000; either could be leased for $6,000 per month.

VERIFICATION OF PERFORMANCE

These findings do not distinguish between the two analyzers. The original study design intended to test the prototype X-MET 940; however, near the end of the data collection at the ASARCO site, a data acquisition problem occurred which prevented any additional use of this analyzer. The demonstration was resumed using an X-MET 920-P. It should also be noted that the first 920-P unit also encountered difficulties with data acquisition and was replaced by the developer. Although both instruments encountered problems, Metorex technical support was responsive to the needs of the demonstration.

The performance characteristics of the X-MET 920-P and 940 include the following:

- **Detection limits:** Precision-based detection limits were determined by collecting 10 replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected MDLs. The results were 120 milligrams per kilogram (mg/kg) or less for arsenic, barium, cadmium, copper, lead, nickel, and zinc. The measured value for chromium was 210 mg/kg. A value for iron was not determined due to insufficient samples in the required concentration range.
- **Throughput:** Average throughput was 10 to 12 analyses per hour using a live count of 240 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.

- **Drift:** This is a measurement of the analyzer's variability in quantitating a known concentration of a standard over time. No data was produced for either analyzer to assess drift.
- **Completeness:** These instruments produced results for 1,192 of the 1,260 samples for a completeness of 94.6 percent, slightly below the demonstration objective of 95 percent. Software and mechanical problems reduced completeness.
- **Blank results:** Lithium carbonate blanks were analyzed periodically throughout the demonstration. Concentrations of copper and iron were detected in all the blanks.
- **Precision:** The goal of the demonstration was to achieve relative standard deviations (RSD) less than 20 percent at analyte concentrations of 5 to 10 times the method detection limits. The RSD values for all analytes were less than 8 percent, except chromium and nickel which had RSD values of 23 and 25 percent, respectively. Values for iron and cadmium were not reported due to insufficient data.
- **Accuracy:** Intramethod accuracy was assessed using site-specific PE soil samples and soil SRMs. The results show that 28 of 38 (73.6 percent) of the analytes in the site-specific PEs were within the quantitative acceptance range of 80 - 120 percent. The barium and cadmium concentrations were underestimated in all PE samples, and one of the three measured values for chromium was overestimated. The soil SRM data showed that 19 of 30 or 63 percent of the analytes were in the acceptable range.
- **Comparability:** This demonstration showed these instruments produced data that exhibited a $\log_{10}$-$\log_{10}$ linear correlation to the reference data. The coefficient of determination (r^2) which is a measure of the degree of correlation between the reference and field data was 0.94 for arsenic, 0.93 for copper, 0.94 for lead, 0.86 for zinc, 0.67 for chromium, and 0.43 for barium. Values for cadmium, nickel, and iron were not reported due to insufficient data.
- **Data quality levels:** Based on precision and comparability to the reference methods, these instruments produced definitive level data for arsenic, lead, copper, and zinc and data of qualitative screening level for chromium and barium. Values for cadmium, nickel, and iron could not be assigned without adequate precision or comparability data.

The results of this demonstration show that either the Metorex X-MET 920-P or X-MET 940 can provide useful, cost-effective data for environmental problem-solving and decision-making. Undoubtedly, these instruments will be employed in a variety of applications, ranging from serving as a complement to data generated in a fixed analytical laboratory to generating data that will stand alone in the decision-making process. As with any technology selection, the user must determine what is appropriate for the application and the project data quality objectives.

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